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Laboratory Feasibility Studies for the Fluidized-Bed Combustion of Spent Potlining from Aluminum Reduction

Battelle Columbus Labs., OH

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LABORATORY FEASIBILITY STUDIES FOR THE FLUIDIZED-BED COMBUSTION OF SPENT POTLINING FROM ALUMINUM REDUCTION

by

B. C. Kim, E. J. Mezey, D. R. Hopper,
A. Wensky, R. Heffelfinger,
and S. E. Rogers

BATTELLE
Columbus Laboratories
505 King Avenue
Columbus, Ohio 43201

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EPA Project Officer:

David C. Sanchez
Industrial Environmental Research Laboratory
Research Triangle Park, North Carolina 27711

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16. ABSTRACT The report gives results of a preliminary assessment of the technical feasibility and environmental acceptability of a fluidized-bed combustion (FBC) process for the disposal of spent potlining waste from the aluminum reduction process. Technical efforts included: (1) differential thermal analyes (DTAs) to establish the operating temperature range to prevent agglomeration in a FBC process, and (2) fixed-bed combustion experiments to determine cyanide destruction and gaseous emissions expected from a FBC process. Test results indicated that FBC should be limited to temperatures below 760 C due to agglomeration of potlining waste that occurs above that temperature. Analysis of the combustion residues showed that 99.9 to 99.99% of cyanide was removed from the potlining and that substantial burning of the carbon in the potlining could be achieved at temperatures as low as 760 C. The results on cyanide removal and carbon combustion are very encouraging and indicate that potential application of a FBC process to the disposal of spent potlining wastes is technically feasible. A small fraction (0.01-2%) of cyanide and a substantial fraction (15-35%) of fluoride were found in the gaseous effluent from combustion of potlining. The gaseous emissions, however, can be adequately controlled and should not pose an insurmountable technical barrier in the FBC process.

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Wastes		Stationary Sources	14G	13H, 07A
Metal Working	Tests	Potlining	11F	14B
Aluminum	Thermal Analysis		07B	
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ABSTRACT

This study was undertaken with the objective of providing a preliminary assessment of the technical feasibility and environmental acceptability of a fluidized-bed combustion process for the disposal of spent potlining waste from the aluminum reduction process. In accomplishing the above, technical efforts were directed to two main areas of laboratory studies: (1) differential thermal analysis experiments to establish the operating temperature range to prevent agglomeration in a fluidized-bed combustion process, and (2) fixed-bed combustion experiments to determine cyanide destruction and gaseous emissions expected from a fluidized-bed combustion process.

The results from the differential thermal analysis and the fixed-bed combustion experiments indicated that the fluidized-bed combustion should be limited to temperatures below 760° C due to agglomeration of potlining waste that occurs above that temperature. Analysis of the combustion residues showed that 99.9 to 99.99 percent of cyanide were removed from the potlining and that substantial burning of the carbon in the potlining could be achieved at temperatures as low as 760° C. The results on cyanide removal and carbon combustion are very encouraging and indicate that potential application of a fludized-bed combustion process to the disposal of spent potlining wastes is technically feasible.

A small fraction (0.01-2 percent) of CN^- and a substantial fraction (15-35 percent) F^- were found in the gaseous effluent from combustion of potlining. The gaseous emissions, however, can be adequately controlled and should not pose an insurmountable technical barrier in the fluidized-bed combustion process. No significant emissions of SO_2 or Na_2O were found during combustion of potlining waste.

Based on the results from the laboratory studies, it is recommended that further investigation of the fluidized-bed combustion process be undertaken through a pilot-plant scale test program. The main technical issue to be addressed in the test program will be related to prove the operability of a fluidized bed over a range of combustion temperatures to accomplish effective destruction of cyanide in the spent potlining waste.

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SECTION 1

INTRODUCTION

Approximately 230,000 tons per year of spent potlining are generated each year in the production of aluminum metal in the U.S. This discarded carbon potlining material contains significant quantities of aluminum and sodium metals, aluminum oxides, fluorides of sodium and aluminum, carbides and nitrides of aluminum, and quantities of cyanide which have penetrated the potliners over their 3 to 5 year (use) life.

Because of the significant energy, chemical and environmental implications of the generation and storage of spent potlining, efforts by the aluminum industry have been directed toward developing environmentally sound resource recovery alternatives. In December 1981, The Environmental Committee of the Aluminum Association, Inc. sponsored a workshop on the storage, disposal, and recovery of spent potlining.

Although in July 1980, spent potlining was listed by EPA as a hazardous waste under RCRA, its listing is currently suspended because of relevant court challenges. However, EPA's interest and responsibility to work with concerned groups in addressing the potlining disposal problem remains active.

The intended scope of this program was to conduct laboratory studies to evaluate the thermal destruction of cyanide in the potlining waste and potential gaseous emissions from fluidized bed combustion of spent potlining. The objective was to assess the technical feasibility and environmental acceptability of the fluidized bed combustion process and provide a basis for recommending and evaluating test plans for follow-up pilot scale fluidized bed combustion of spent potlining.

SECTION 2

CONCLUSIONS

The results of laboratory studies on the combustion of spent potlining from the aluminum reduction process have identified agglomeration as a potential problem for a fluidized-bed combustion process due to softening or melting of the potlining during the combustion process. Although the combustion residue (ash) from the potlining could be maintained free flowing without agglomeration at temperatures above 900° C using a kaolin clay as an additive, the potlining itself was observed to agglomerate at temperatures as low as 760° C even with the use of the clay additive. Whether the turbulent mixing obtained in the fluidized-bed combustion process could overcome the agglomeration problem appears to be the key technical issue that must be resolved to prove the fluidized-bed combustion process for application in spent potlining disposal.

The results from fixed-bed combustion experiments have also indicated that 99.9 to 99.99 percent of the cyanide in the potlining can be removed at 760° C. Small quantities of HCN were found in the gaseous combustion products from fixed-bed combustion tests performed on potlining samples. It is expected that the HCN generated during combustion of potlining waste can be destroyed by providing sufficient gas-phase residence time at combustion temperature in a fluidized-bed combustor. No detectable emission of SO_2 was found in the flue gas from combustion of potlining.

A significant evolution of HF has been found from combustion of potlining. Therefore, the flue gas from potlining waste combustion will need to be treated to control HF emission and possibly reclaim the fluoride value.

Results from fixed-bed combustion experiments have indicated that the carbon in potlining waste can be ignited and burned efficiently at temperatures as low as 760° C. The reactivity of the carbon in spent potlining waste appears to be sufficiently high to obtain a high combustion efficiency (> 90%) and possibly sustain combustion without an auxiliary fuel in a fluidized-bed combustor.

No significant volatilization of Na_2O from possible reactions between potlining and CaO or water vapor was detected at 760° C. Therefore, generation of Na_2O from combustion of potlining and associated slagging and fouling problems are not expected in the fluidized-bed combustion process.

SECTION 3

RECOMMENDATIONS

Based on the findings from the laboratory feasibility studies, it is recommended that a fluidized-bed combustion test program be undertaken. The main technical issue to be addressed in a fluidized-bed test program will be to demonstrate operation of a fluidized bed over a practical range of temperature to accomplish effective destruction of cyanide without bed agglomeration. The target temperature range should be at least 50° C (and preferably 100° C) to allow for normal temperature fluctuations and process upsets that can be anticipated in a full-scale fluidized-bed combustion unit. The clinkers formed by molten potliner waste are extremely hard, and formation of such clinkers in a fluidized bed by temperature excursions would cause a major shutdown and possibly extensive damage to the refractory in a fluidized-bed combustor. Should the bed agglomeration cause a severe problem in fluidized bed operation, other combustion technologies, such as a rotary kiln incinerator, should be considered as an alternative approach.

Additional recommendations addressed to a fluidized-bed test program are listed below:

1. Due to the inhomogeneous nature of potlining waste, multiple sampling of the feed and ash during the test program is recommended.
2. The suggested range of combustion temperature to be investigated is 600 to 800° C. The use of kaolin clay as an additive should be considered to increase combustion temperature as needed.
3. Flue gas analysis should focus on HCN and HF emissions to further establish the need for downstream treatment of the flue gas. The level of HF found in the flue gas has an important bearing on possible recovery of fluoride value and waste heat.
4. Cyanide destruction by the fluidized-bed combustion should be confirmed by performing leaching tests on the combustion residue.

SECTION 4

LABORATORY STUDIES

The laboratory studies were conducted in two parts consisting of (1) differential thermal analysis experiments to establish the operating temperature for the fluidized-bed combustion process dictated by ash agglomeration and (2) fixed-bed combustion experiments to determine cyanide destruction and gaseous emissions at the operating temperature established in the experiments of(1).

POTLINING SAMPLES

Potlining samples were supplied by Aluminum Company of America (Alcoa) and Reynolds Metals Company. Chemical analyses of the potlining samples are shown in Table 1. Both of these samples exhibited a strong odor of ammonia, probably produced by hydrolysis reaction between moisture in ambient air and aluminum nitride present. The samples contained some coarse (approximately 1/4-inch size) particles and appeared nonhomogeneous. Therefore, the samples were dumped from the shipping containers, mixed, and coned and then split into smaller aliquots by sequential quartering and coning or by using a sample splitter. The head samples prepared in this manner were the 1/32 cut of the Alcoa sample and the 1/128 cut of the Reynolds sample. The head samples were pulverized and stored in sealed containers for use in the DTA studies.

DIFFERENTIAL THERMAL ANALYSIS STUDIES

Differential thermal analysis (DTA) experiments were carried out on the two potlining samples and the ash samples prepared from the potlining samples. Ash samples were prepared by burning off the combustible matter from the potlining samples in a muffle furnace in ambient air at 750° C to constant residue weight. DTA experiments were also carried out on mixtures of the potlining ash samples with a

TABLE 1. COMPOSITIONS OF POTLINING SAMPLES[a]

	COMPOSITION, PERCENT	
	ALCOA	REYNOLDS
C	28.6	42.7
Al	14.2	4.7
Si	4.76	0.11
Fe	3.23	0.27
Na	13.9	20.0
F	18.2	17.6
CN	0.09	0.135
Al Carbide	0.46	-
Ash	68.7	57.1

(a) Analyses provided by Alcoa and Reynolds except ash analyses performed by Battelle

reagent-grade calcium oxide or a commercial-grade kaolin clay used as additives. Composition of the clay is given in Table 2. DTA scans were made at 50 C/min under flowing dry air atmosphere. Zinc and K_2CrO_4 standards were used to calibrate the temperature scale. Results from the DTA experiments are shown in Figures 1 through 14.

Figure 1 is a DTA scan of the reagent grade CaO. Two endotherms were observed: The first at 310 C due to dehydration and the second at 605° C due to carbonate decomposition. The DTA scan shown in Figure 2 was made on the clay that had been preheated in an oven for 2 hours at 750° C to remove moisture from the sample. The scan shows a large exotherm of an unknown origin in the 200-400° C range and a broad nondescript endotherm in 550-900° C range.

DTA scans of the Reynolds potlining are shown in Figure 3. The DTA scans were run first on the potlining sample (labeled Run 1 on the figure) and the ash sample (labeled Run 2 on the figure) resulting from the same potlining sample. The potlining sample in Run 1 exhibited an endotherm in the 30-210° C range, indicating an initial phase change the potlining undergoes at a low temperature. The potlining further exhibited two additional endotherms of unknown origins at approximately 390° C and 830° C. These two endotherms were superimposed on the large exotherm observed between 310 and 900° C which was probably due to the carbon burning reaction.

Addition of kaolin clay has been shown to be effective in preventing stickiness in fluidized beds containing sodium salts with low melting points.[1]

DTA results on the ash prepared from the Reynolds potlining waste are shown in Figures 4 through 8. DTA scan on the ash by itself without any additive is shown in Figure 4. Following each DTA run on an ash sample, the same ash sample was heated to the temperatures at which endotherms were observed in the DTA scan to determine the temperature at which the ash starts to agglomerate. On the Reynolds potlining ash sample, the agglomeration occurred between the first and the second endotherms observed at 713° C and 804° C, respectively. Although the Reynolds potlining undergoes a phase change at a relatively low temperature below 210° C as indicated earlier, the ash becomes loose as combustion proceeds and remains loose up to the indicated agglomeration temperature range. This type of behavior is also observed with other solid fuels, such as bituminous coal, petroleum pitch and

TABLE 2. MANUFACTURER'S DATA ON KAOLIN CLAY

Product Discription: No. 6 Tile Clay[(a)]

Manufacturer: Georgia Kaolin Company, Inc.
433 North Broad Street
Elizabeth, N. J. 07207

CHEMICAL COMPOSITION

Component	Percent by Weight
SiO_2	46.9
Al_2O_3	38.2
Fe_2O_3	0.35
TiO_2	1.42
CaO	0.43
MgO	0.58
Na_2O	0.04
Loss on Ignition at 1000 C	13.9

(a) Quoted price is $63.50/ton in bulk quantities.

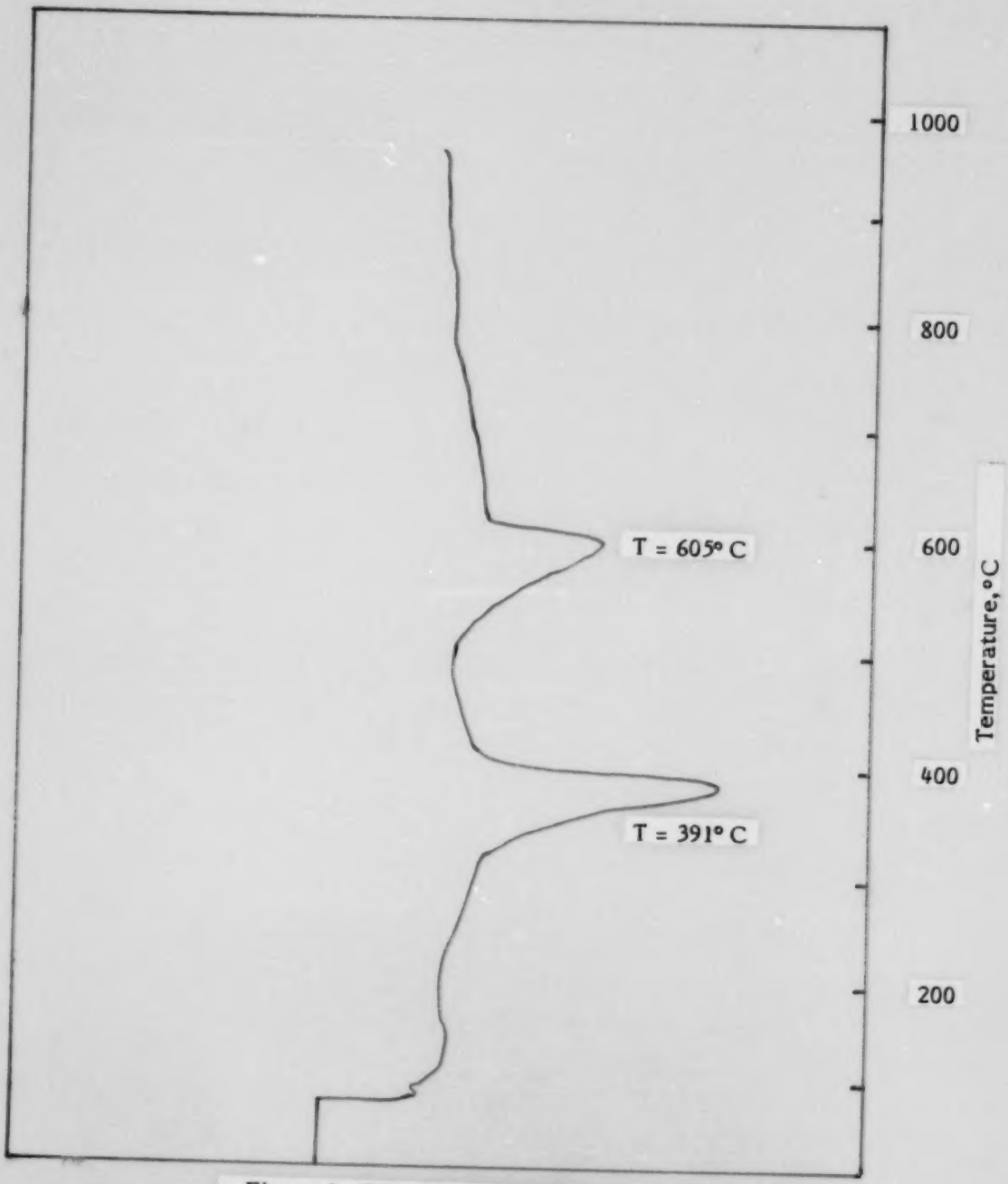

Figure 1. DTA Scan of CaO.

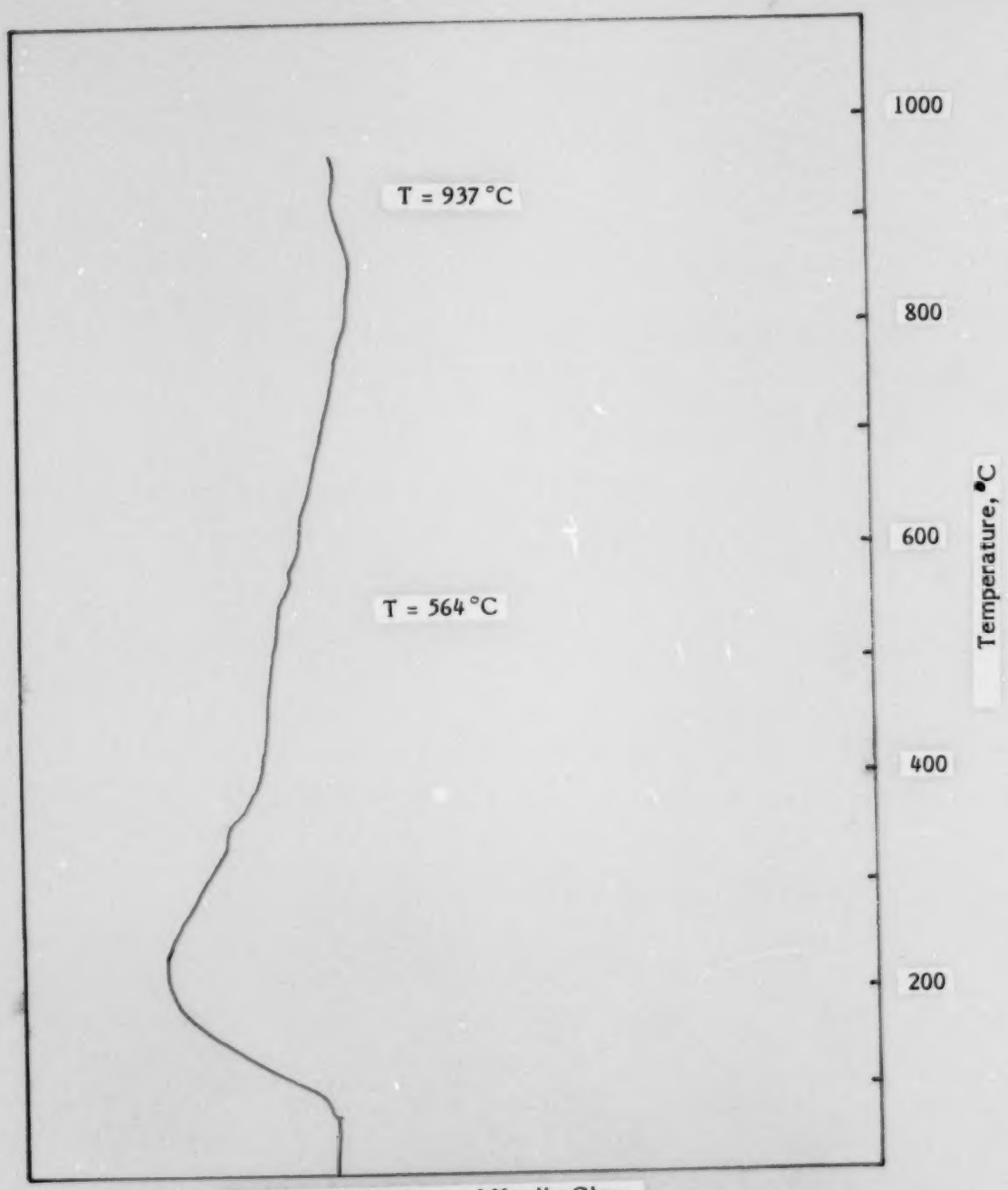

Figure 2. DTA Scan of Kaolin Clay.

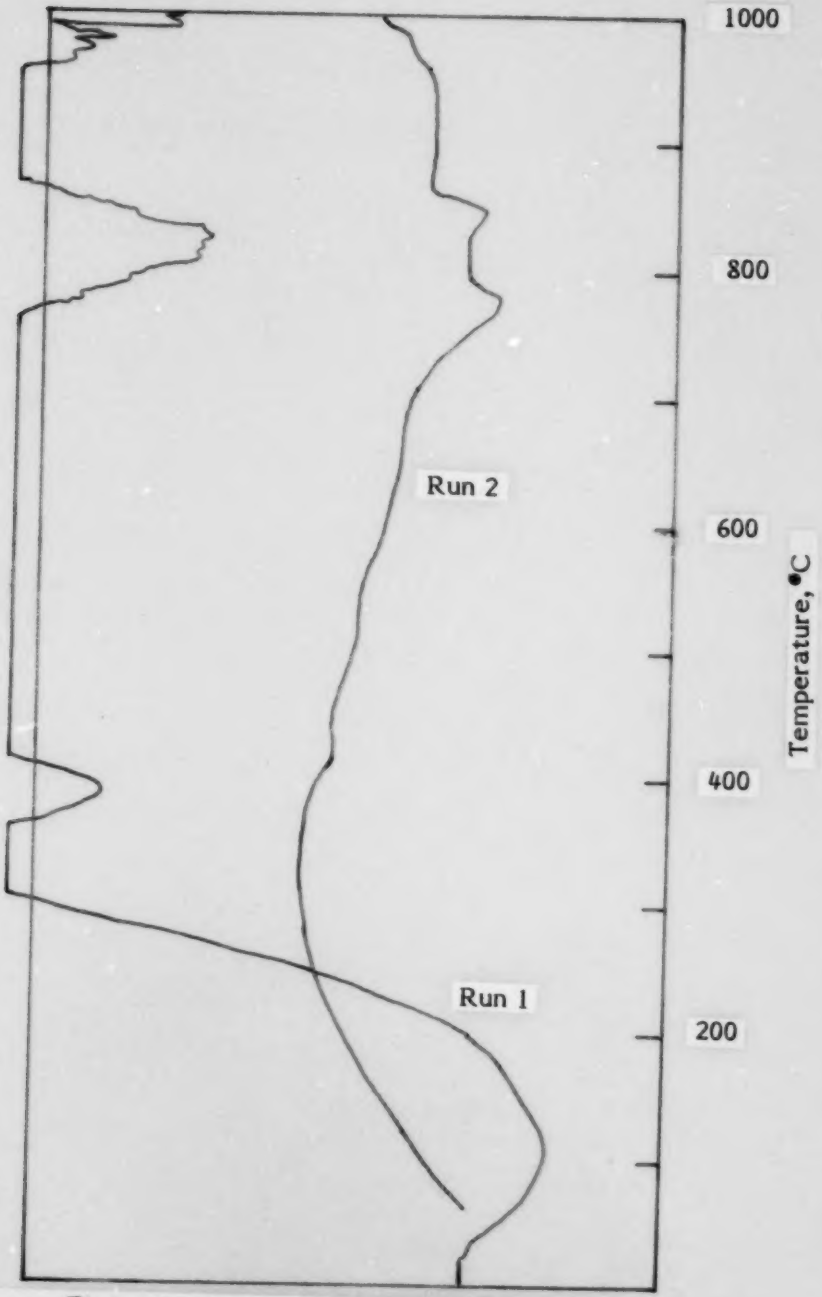

Figure 3. DTA Scan of Reynolds Potlining.

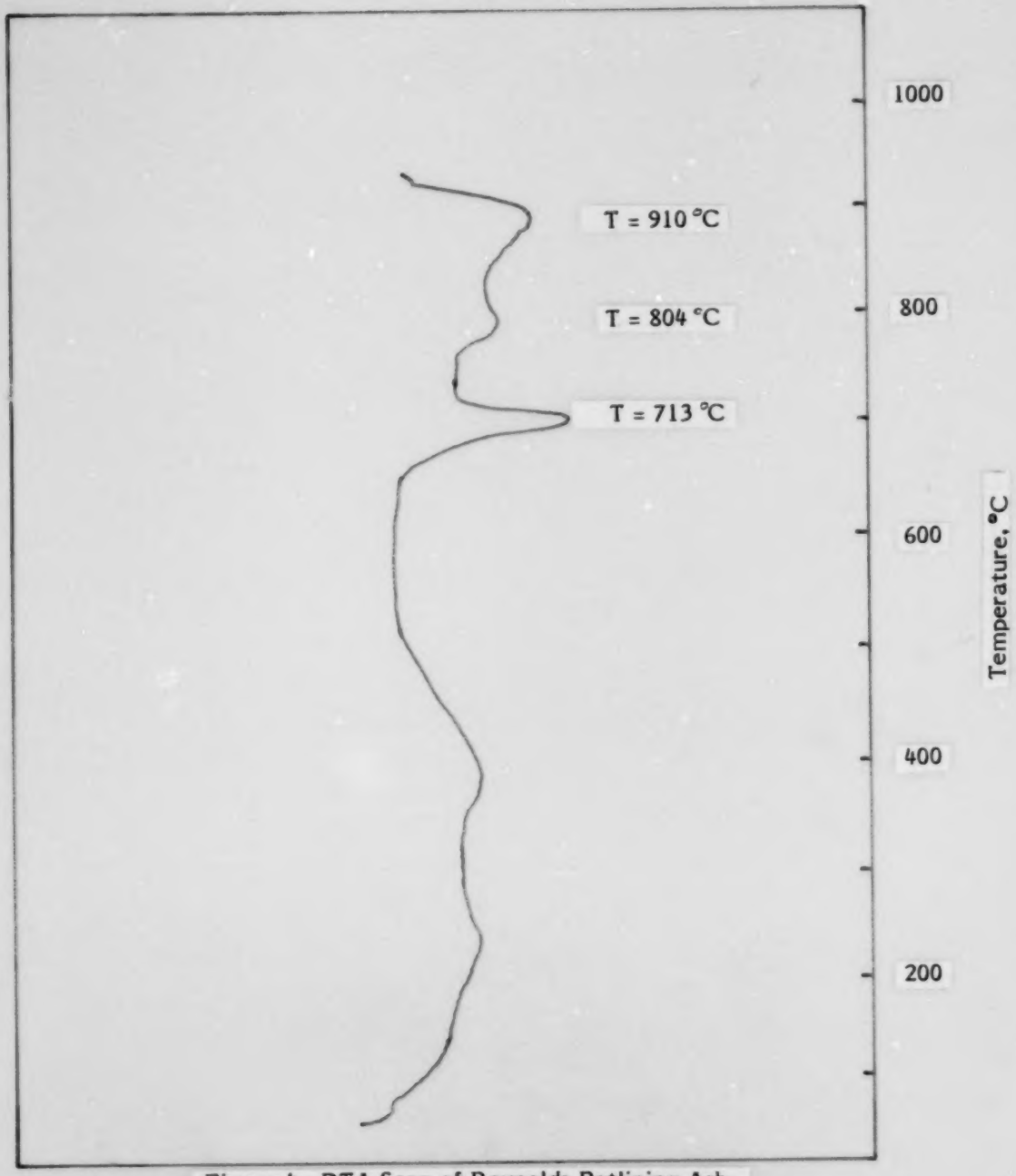

Figure 4. DTA Scan of Reynolds Potlining Ash.

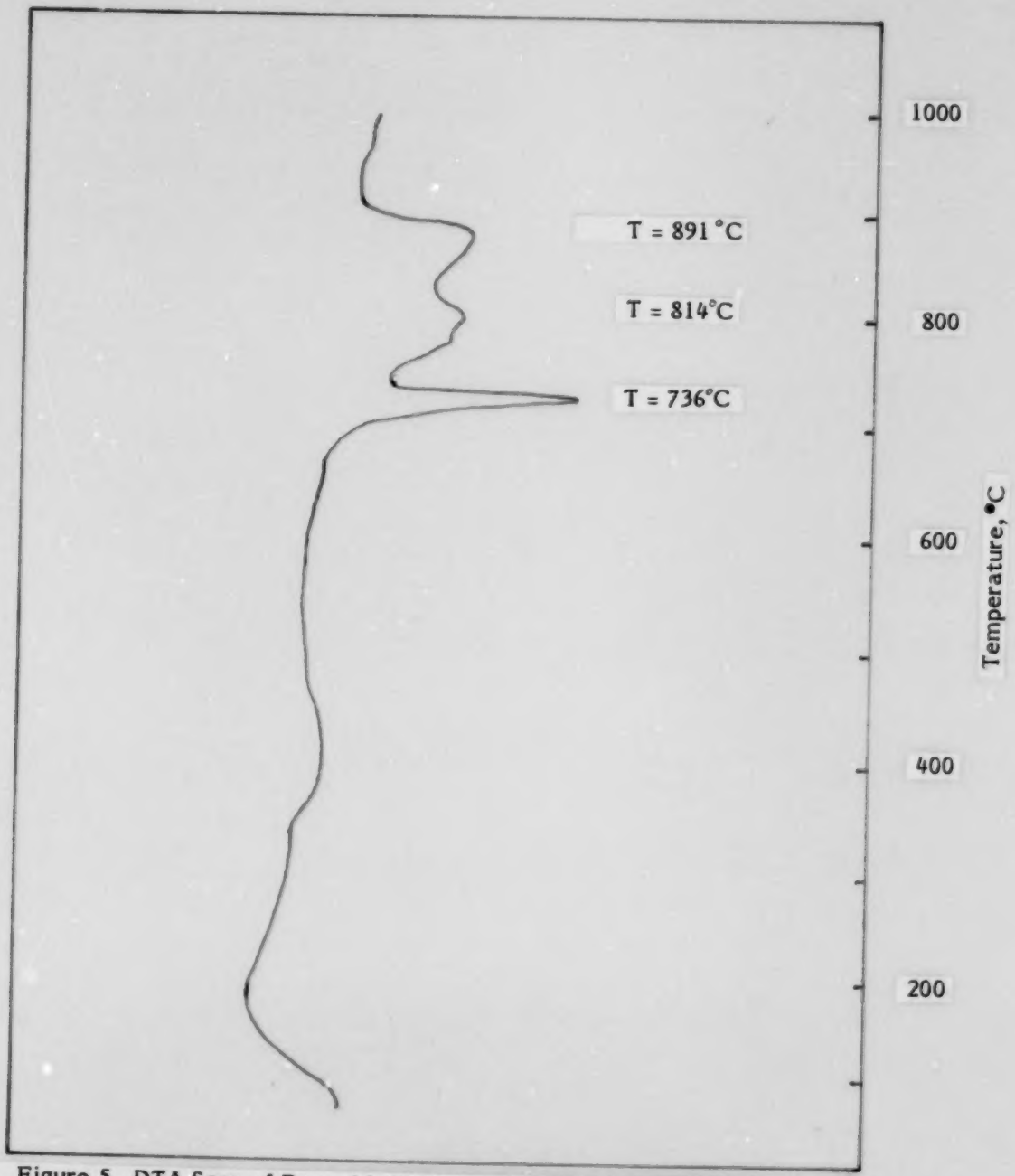

Figure 5. DTA Scan of Reynolds Potlining Ash Mixed with 5.6 Percent CaO.

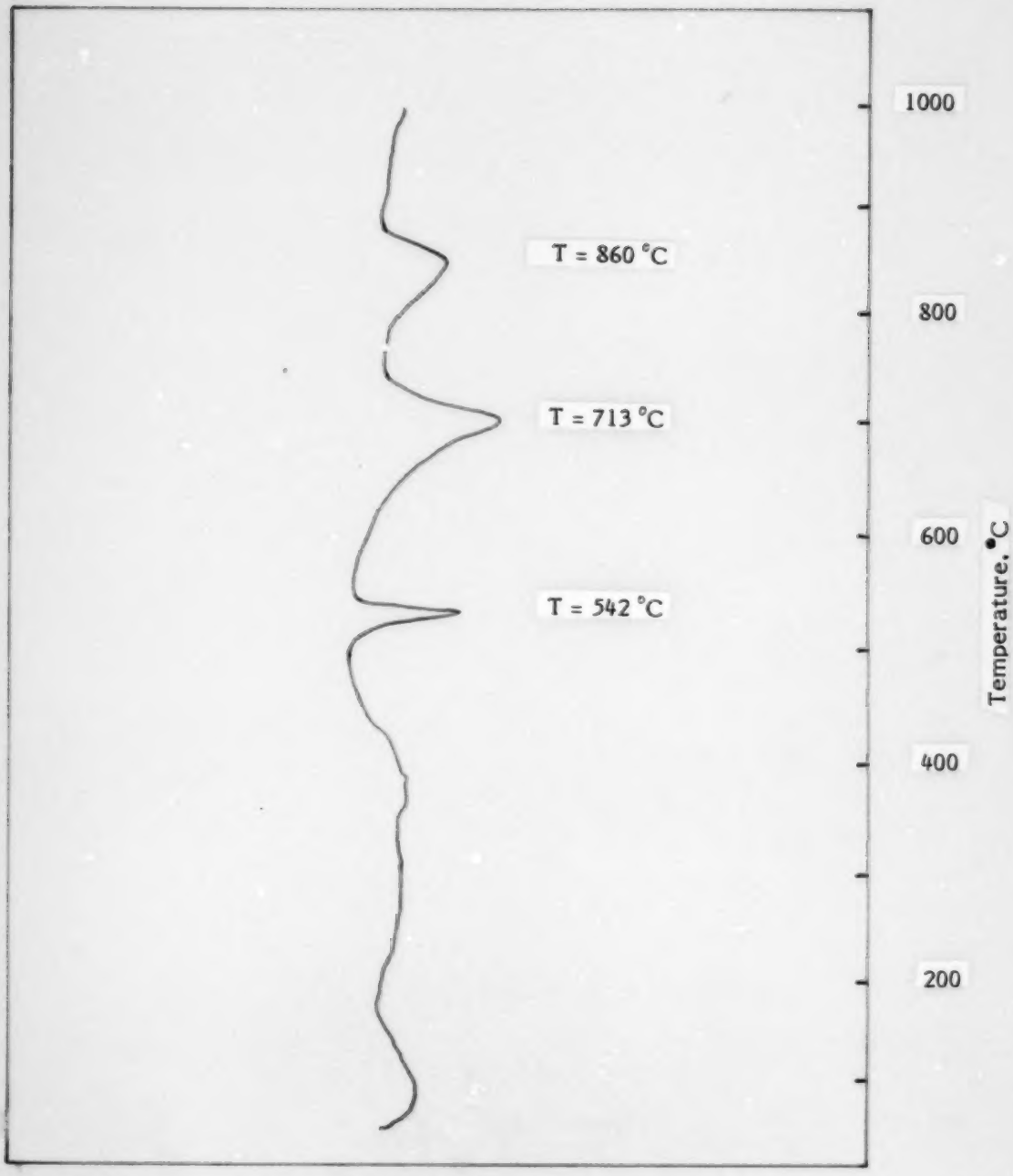

Figure 6. DTA Scan of Reynolds Potlining Ash Mixed with 56 Percent CaO.

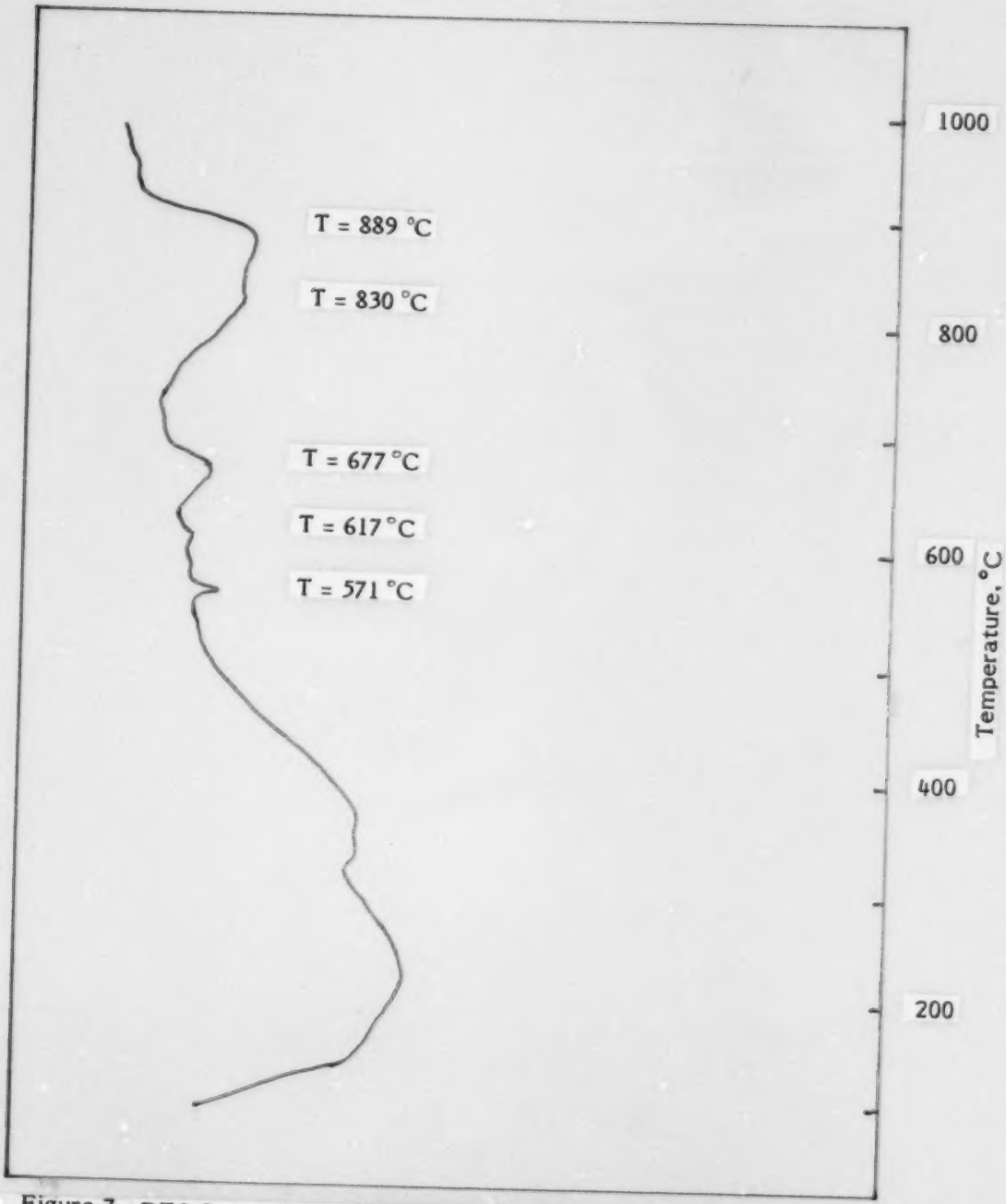

Figure 7. DTA Scan of Reynolds Potlining Ash Mixed with 10 Percent Kaolin Clay.

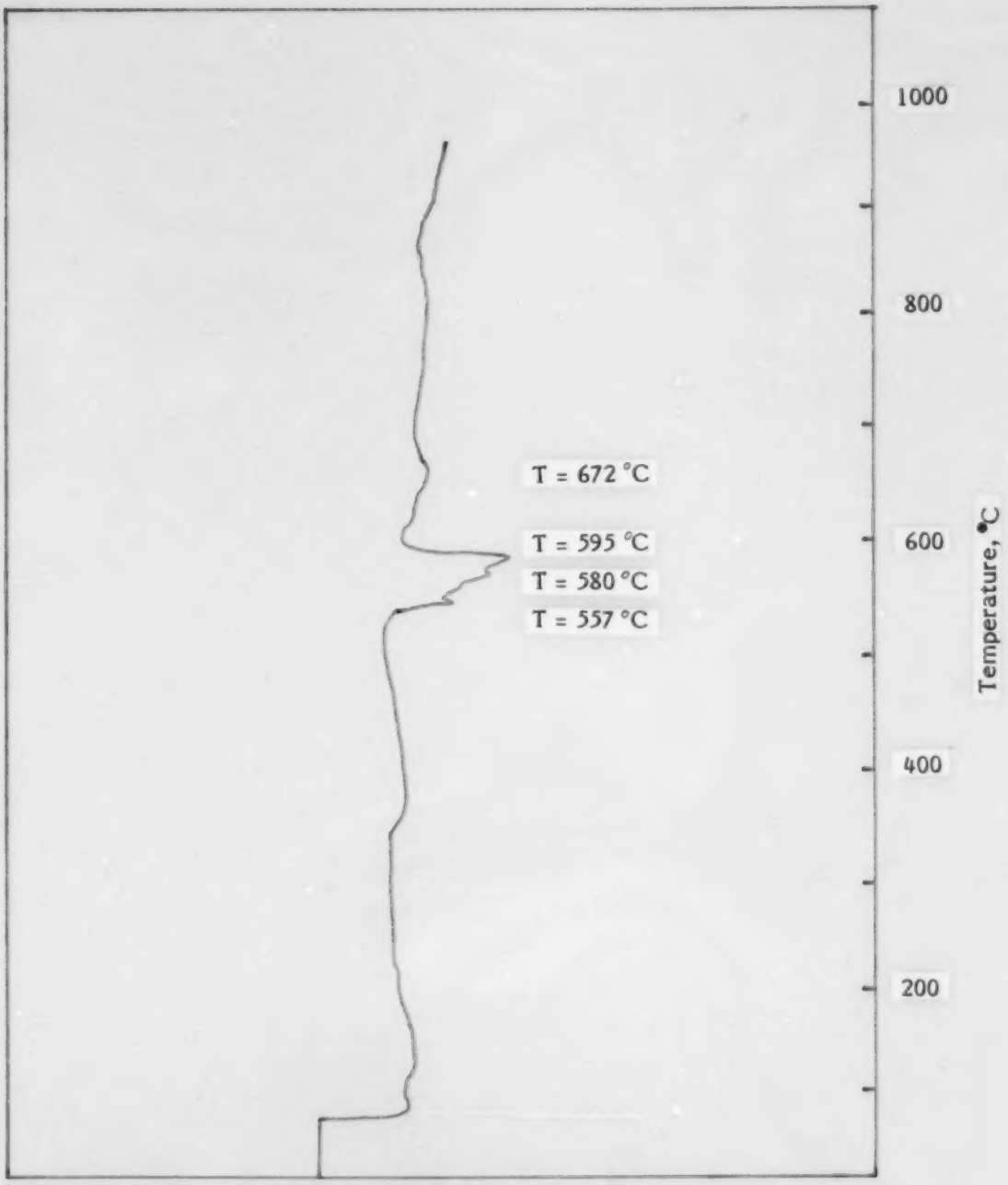

Figure 8. DTA Scan of Reynolds Potlining Ash Mixed with 100 Percent Kaolin Clay.

plastics, that undergo an initial softening or melting stage below combustion temperatures. These fuels can be burned in a fluidized bed, provided the initial softening or melting of the fuels is transitory.

Calcium oxide and a kaolin clay were tested as additives for raising the ash agglomeration temperature. DTA scans on the Reynolds potlining ash with either CaO or clay addition are shown in Figures 5 through 8. Subsequent ash agglomeration tests showed the ash agglomeration temperatures to be: (1) 736-815° C with 5.6 percent CaO addition (based on 0.1 g $CaCO_3$/g potlining ash), (2) 542-713° C with 56 percent CaO addition (based on 1 g $CaCO_3$/g ash), (3) 630-889° C with 10 percent clay addition (0.1 g clay/g ash), and (4) above 900° C with 100 percent clay additon (1 g clay/g ash). These results indicate that CaO addition has little or adverse effect on ash agglomeration and that the clay addition increases ash agglomeration temperature.

Results from the DTA experiments performed on the Alcoa potlining and ash sample are shown in Figures 9 through 14. The DTA scans of the Alcoa potlining sample are shown in Figure 9. The first scan, labeled Run 1 on Figure 9, was performed on the potlining sample. This scan showed a large exotherm in the 400-900° C range due to the carbon combustion reaction. The Alcoa potlining did not exhibit the low-temperature phase change that was observed with the Reynolds potlining. The DTA scan of the Alcoa potlining confirmed this finding by the absence of an endotherm characteristic of the melting process at low temperatures. The second DTA scan labeled Run 2 in Figure 9, was made on the residue resulting from Run 1. The second scan on the residue showed small endotherms at 510 C, 660° C, and 860° C.

DTA scans on the Alcoa potlining ash are shown in Figure 10 through 14. The DTA scan on the ash sample with no additive showed endotherms at 561° C, 726° C, 798° C, and 869° C. Subsequent ash agglomeration tests showed the ash agglomeration temperature to be in the 798-869° C range. DTA scans on the Alcoa potlining ash with either CaO or clay addition are shown in Figures 11 through 14. Subsequent ash agglomeration tests performed on these samples showed the ash agglomeration temperatures to be: (1) 730-860° C with 5.6 percent CaO additon (based on 0.1 g $CaCO_3$/g potlining ash), (2) 736-815° C with 56 percent CaO addition (based on 1 g $CaCo_3$/g ash), (3) 630-889° C with 10 percent clay addition (based on 0.1 g clay/g

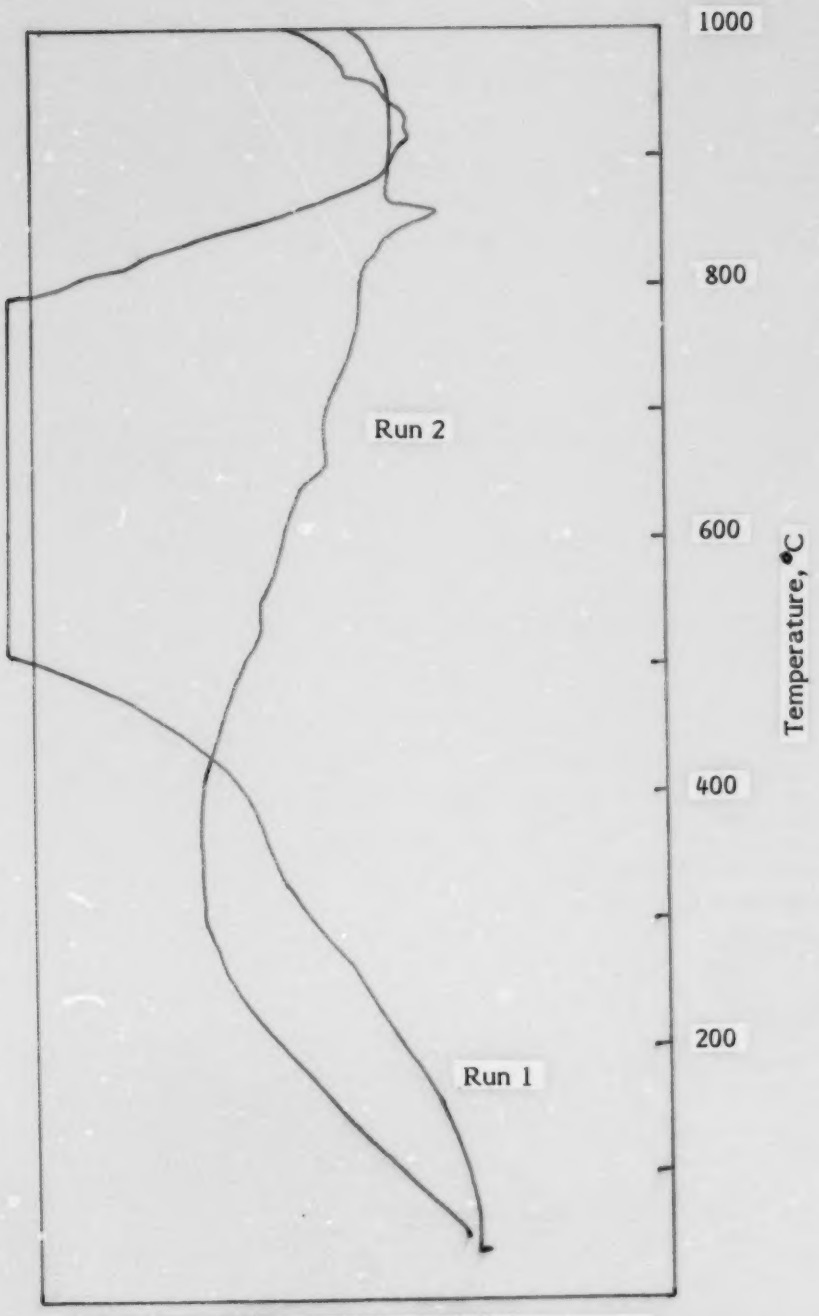

Figure 9. DTA Scan of Alcoa Potlining.

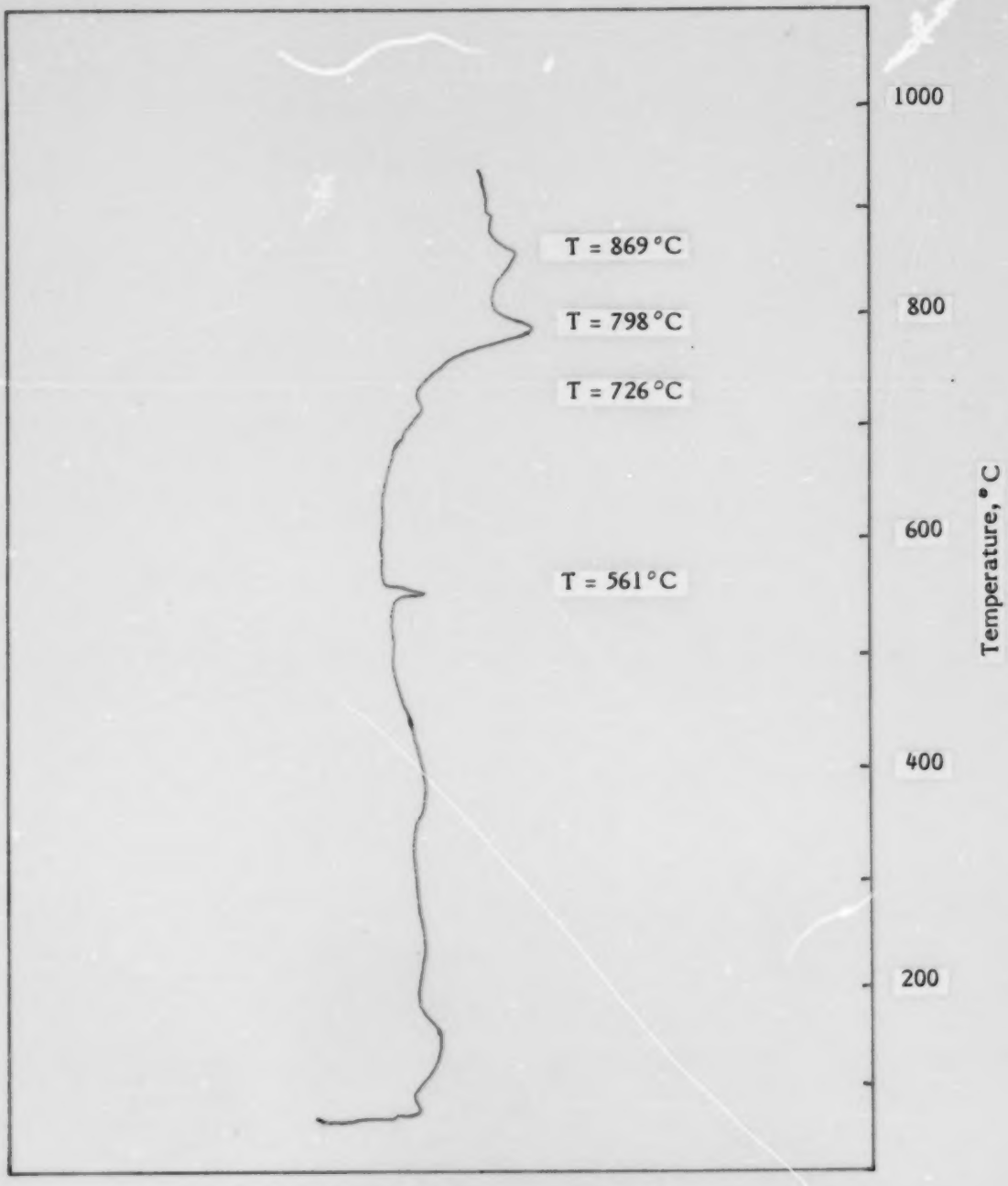

Figure 10. DTA Scan of Alcoa Potlining Ash.

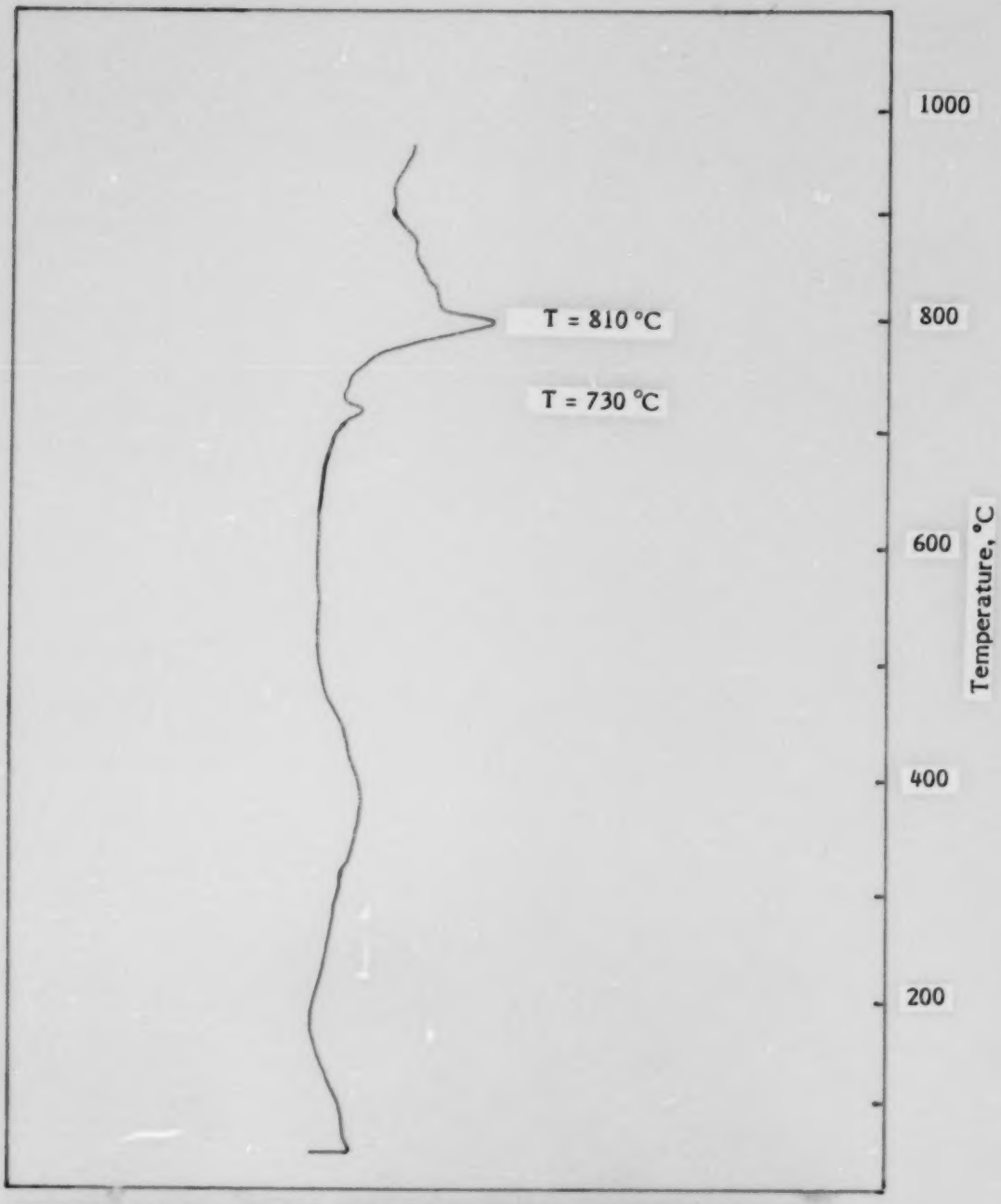

Figure 11. DTA Scan of Alcoa Potlining Ash Mixed with 5.6 Percent CaO.

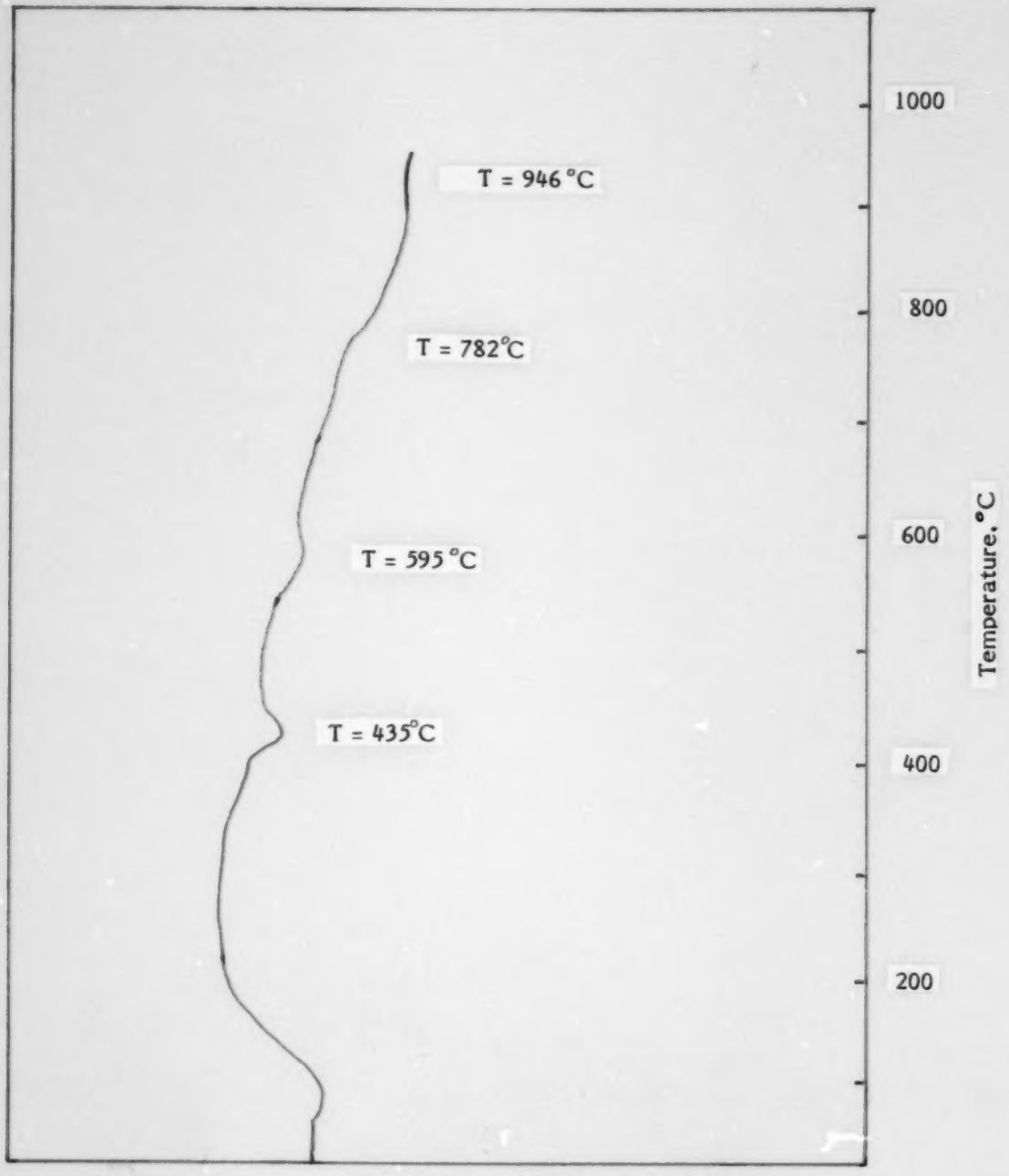

Figure 12. DTA Scan of Alcoa Potlining Ash Mixed with 56 Percent CaO.

Figure 13. DTA Scan of Alcoa Potlining Ash Mixed with 10 Percent Kaolin Clay.

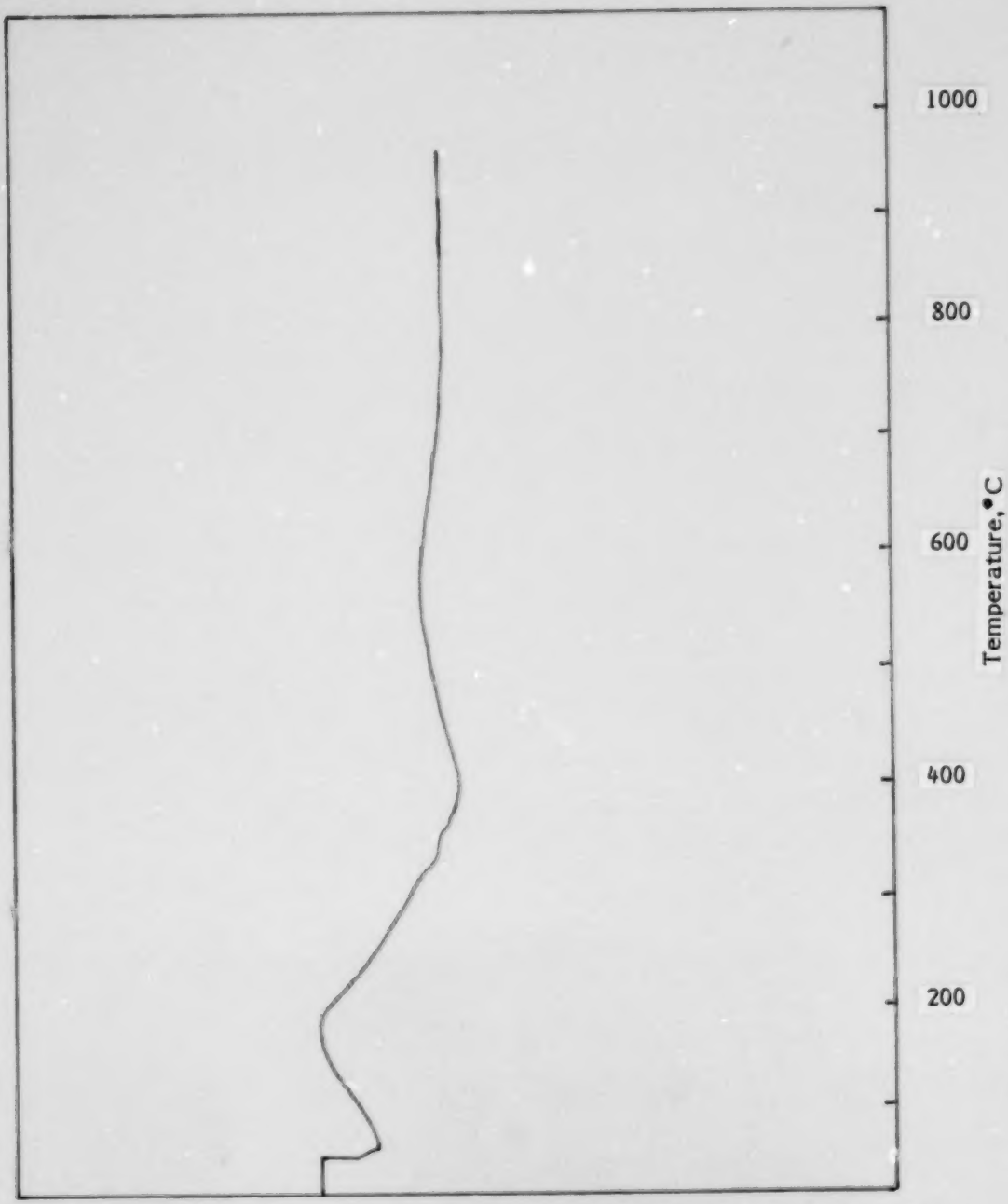

Figure 14. DTA Scan of Alcoa Potlining Ash Mixed with 100 Percent Kaolin Clay.

ash), and (4) above 900° C with 100 percent clay addition (based on 1 g clay/g ash). These results indicate little or adverse effect of CaO addition and a beneficial effect of clay additon on raising the ash agglomeration temperature.

Results from the ash agglomeration tests are presented in Table 3 and summarized below:

- Alcoa potlining ash has higher agglomeration temperature than Reynolds potlining ash
- Addition of CaO has little or no adverse effect on raising ash agglomeration temperature
- Addition of clay results in significant increase in ash agglomeration temperature to above 900° C.

FIXED-BED COMBUSTION STUDIES

Fixed-bed combustion experiments were performed to determine cyanide destruction and emissions of cyanide and fluoride in the gaseous combustion products. The experimental apparatus is shown in Figure 15. The reactor was made of a quartz tube, 2 inches ID by 44 inches long. A frit was installed approximately 10 inches from the bottom of the reactor to serve as a bed support. A bed of approximately 2-inches of alumina chips were placed on the frit to serve as a protective layer against possible attack of the frit by potlining. The flow of combustion air was downward as indicated on the figure. The reactor was heated by an electric tubular furnace. The off-gas from the reactor was first cooled in a water bath and split into two streams: one stream directed to an impinger train loaded with 0.1 N NaOH solution for collection of HCN and HF and the other stream directed to on-line gas analyzers for measurements of CO, CO_2, SO_2 and O_2.

To perform a combustion test, the furnace was preheated to a prescribed temperature. The reactor was loaded with a potlining sample and then inserted into the furnace and quick connections were made on the reactor inlet and outlet ports. Dry combustion air was supplied from a compressed air cylinder. During the test, the air feed rate to the reactor was controlled at 7 liters/min and the total flue-gas flow through the impinger train was measured with a wet test meter. Flue gas

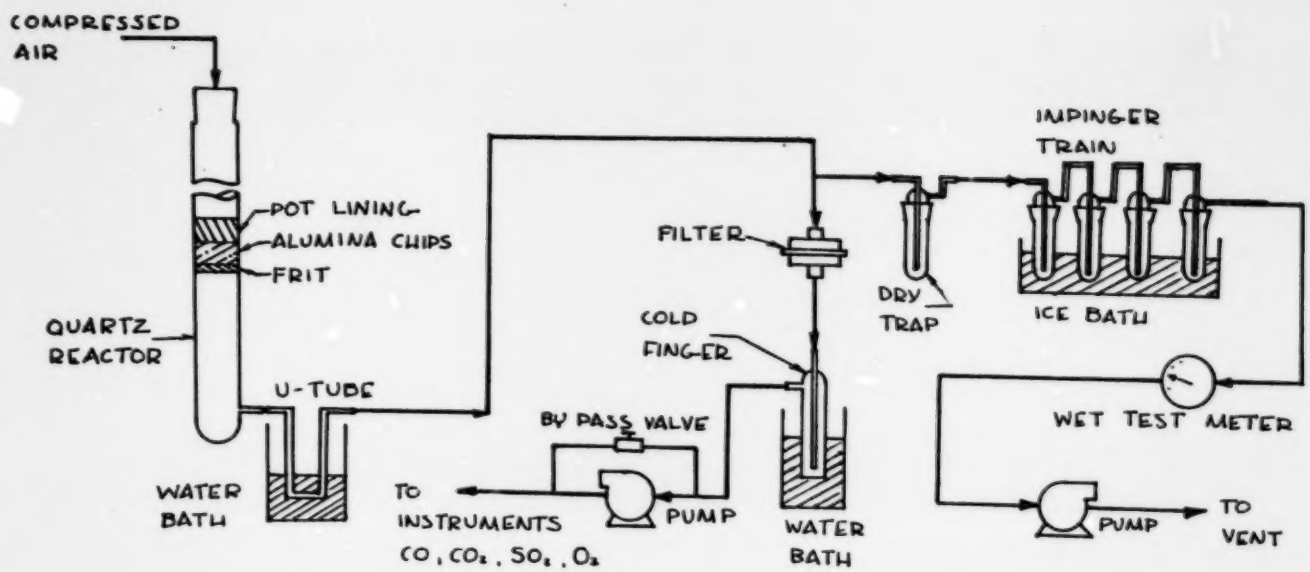

Figure 15. Schematic of Apparatus used in Fixed-Bed Combustion Tests.

compositions were recorded continuously on strip chart recorders. After the test, the combustion residue in the reactor and liquid collected in the traps were removed and submitted to Battelle's analytical laboratory for chemical analyses. Parameters investigated in the fixed-bed combustion tests were as follows:

1. Potlining sample quantity: 12 and 75 grams
2. Additives: clay addition at 15 to 87 percent based on ash in the potlining sample, and CaO additon at 87 percent based on ash in the potlining sample
3. Temperature: 760 to 911° C
4. Residence Time: 30 and 120 minutes
5. Water vapor content in combustion air feed: Approximately 2.6 percent in one test.

A significant finding from the fixed-bed combustion tests was the lower agglomeration temperature of the potlining samples compared with the agglomeration temperatures of the potlining ash samples observed in the prior DTA and ash agglomeration tests. Results on potlining agglomeration observed in the fixed-bed combustion tests are summarized in Table 3. The prior DTA and ash agglomeration tests conducted with clay addition had indicated ash agglomeration temperatures above 900° C for both Alcoa and Reynolds potlining ash samples. The potlining samples in the fixed-bed combustion tests showed agglomeration at temperatures as low as 760° C. One possible cause of agglomeration of potlining is sintering during dehydration of the $Al(OH)_3$ that is formed by hydrolysis of AlN in potlining. Under this hypothesis, agglomeration could be avoided in a fluidized bed, since dehydration of $Al(OH)_3$ is commercially practiced in fluidized-bed calciners. However, the probability of $Al(OH)_3$ sintering process to be the sole cause of agglomeration is remote in view of the complex salts contained in the potlining compositions. Therefore, agglomeration of potlining poses a potential problem in a fluidized-bed combustion process. Results from the fixed-bed combustion tests on bed agglomeration are summarized below:

1. The Alcoa potlining sample showed greater tendency for agglomeration than the Reynolds potlining sample. (Compare Tests 5 and 9, or Tests 7 and 8). This behavior of the potlining samples is opposite from

TABLE 3. SUMMARY OF FIXED-BED COMBUSTION TESTS ON POTLINING SAMPLES

Test	Potlining		Clay Addition		Temperature, °C	Residence Time, Min.	Results
	Type	Sample Wt., g	Wt., g	Percent of Ash[a]			
1	Alcoa	75	7.5	15	911	30	Very severe agglomeration; quartz reactor cracked apparently caused by fused potlining
2	Alcoa	75	7.5	15	762	120	Very severe agglomeration
3	Reynolds	75	7.5	17	760	120	Very severe agglomeration
4	Reynolds	75	37.5	87	762	30	Very severe agglomeration
5	Reynolds	12	6	87	760	120	Slight agglomeration
6	Alcoa	12	4.1	50	875	30	Very severe agglomeration
7	Reynolds	12	6	87	760	30	Slight agglomeration
8	Alcoa	12	7.2	87	760	30	Severe agglomeration
9	Alcoa	12	7.2	87	760	120	Severe agglomeration
10	Reynolds	12	6+6 (CaO)	87+87(CaO)	760	120	Slight agglomeration
11[b]	Reynolds	12	6	87	760	120	Slight agglomeration

(a) Based on 68.7 percent ash in the Alcoa potlining and 57.1 percent ash in the Reynolds potlining.

(b) Combustion air was humidified to approximately 2 percent water vapor concentration.

the behavior observed with the ash samples in the DTA tests which showed higher agglomeration temperatures for the Alcoa sample compared with the Reynolds sample.

2. Reducing the sample size decreased the apparent extent of agglomeration of potlining. (Compare Tests 4 and 7). However, even a smaller quantity of test samples showed agglomeration at 760° C (Tests 8 and 9).

Due to the severe agglomeration of the bed that resulted in poor gas-solid contact, the ash residue and impinger train samples that were selected for analyses were chosen from those tests performed during the latter part of the combustion test program in which bed agglomeration was substantially reduced by decreasing the combustion temperature and the amount of potlining sample used. Material balance results describing the fate of cyanide, fluorine, carbon and sodium during the fixed-bed combustion of potlining were calculated from these sample analyses results.

Tests 5, 7, 8 and 9 presented in Tables 4 through 6 were performed to determine cyanide destruction, emissions of cyanide and fluoride in flue gas, and carbon combustion efficiency. Results can be summarized as follows:

1. Cyanide removal from the potlining in the range of 99.9 to 99.99 percent was achieved with both Alcoa and Reynolds potlining samples at 760° C and 30 to 120 minutes residence time. Small quantities of cyanide in the range of 0.01 to 2 percent of feed were found in the flue gas. It is expected that a fluidized-bed combustor should provide sufficient gas-phase residence time in the bed and in the freeboard to destroy the cyanide evolved as HCN from the potlining.
2. Approximately 20 to 30 percent loss of fluoride from potlining was observed under the conditions employed in the combustion tests. Fluoride collection from the flue gas was measured to be in the range of 0.2 to 8.7 percent. The fluoride unaccounted for was probably lost by reaction with the quartz reactor and other glass surfaces that came in contact with the flue gas. Nevertheless, the results strongly suggest a significant fraction of fluoride is generated as HF from

TABLE 4. MATERIAL BALANCE ON CYANIDE

Test	Potlining Type	Temperature, °C	Residence Time, min	CN In, 10^{-6}g	CN Out, 10^{-6}g		CN Out, percent of feed	
				Potlining	Ash	Flue Gas	Ash	Flue Gas
7	Reynolds	760	30	16,000	15.6	67	0.1	0.4
5	Reynolds	760	120	16,000	6.9	296	0.04	2
8	Alcoa	760	30	11,000	1.3	1	0.01	0.01
9	Alcoa	760	120	11,000	1.5	71	0.01	0.6

TABLE 5. MATERIAL BALANCE ON FLUORIDE

Test	Potlining Type	Temperature, °C	Residence Time,min	F In,g Potlining	F Out, Ash	F Out, Flue Gas	F Out, percent of feed Ash	F Out, percent of feed Flue Gas
7	Reynolds	760	30	2.11	1.76	0.004	83.4	0.2
5	Reynolds	760	120	2.11	1.66	0.017	78.7	0.8
8	Alcoa	760	30	2.18	1.68	0.059	77.1	2.7
9	Alcoa	760	120	2.18	1.40	0.19	64.2	8.7

TABLE 6. MATERIAL BALANCE ON CARBON

Test	Potlining Type	Temperature, °C	Residence Time, min	C In, g	C Out, g		C Out, percent of feed	
				Potlining	Ash	Flue Gas	Ash	Flue Gas
7	Reynolds	760	30	5.12	1.5	4.9	29	96
5	Reynolds	760	120	5.12	0.74	6.1	15	120
8	Alcoa	760	30	3.43	1.0	3.5	29	100
9	Alcoa	760	120	3.43	0.08	4.2	2	122

potlining during the combustion process. Therefore, the flue gas should be treated to control HF emission and possibly recover the fluoride value.

3. Results on carbon balance show approximately 70 to 90 plus percent carbon combustion efficiencies in the combustion tests. The total carbon found in the ash and the flue gas was found to be approximately 25 to 35 percent above the carbon introduced with the potlining, indicating a reasonable closure on the carbon balance. Combustion efficiency in a fluidized bed is expected to be higher due to improved mixing and gas-solid contact in a fluidized bed. Therefore, combustion of potlining in a fluidized bed at temperatures as low as 760° C appears to be feasible
4. No detectable emission of SO_2 was found in the flue gas from combustion of potlining.

Tests 10 and 11, presented in Table 7, were performed to determine volatilization of Na_2O by reactions of a potlining sample with CaO and with H_2O vapor. The tests were carried out with the Reynolds potlining sample that has a higher Na content than the Alcoa potlining sample. Test 10 was carried out with CaO addition and dry combustion air. Test 11 was carried out with the combustion air humidified to approximately 2 percent water vapor. Results on sodium balance show essentially no volatilization of Na_2O either due to CaO or H_2O vapor under the conditions employed in the combustion tests.

QUALITY ASSURANCE

Before the sampling and analysis procedures were initiated a quality assurance plan was submitted to the EPA project officer. This plan addressed specific elements of quality control applied to this study in addition to general quality assurance measures that are routinely performed at Battelle to ensure defensibility of analytical results, e.g., periodic instrument audits, defined calibration procedures, and unannounced audits by one of Battelle's QA officers.

A Premco Model 150 Differential Thermal Analysis Instrument was used to determine possible ash agglomeration temperatures. During the fixed-bed combustion experiments, the entire effluent was first directed into a quartz sample manifold.

TABLE 7. MATERIAL BALANCE ON SODIUM

Test	Potlining Type	Temperature, °C	Residence Time,min	Additive	Na In,g Potlining	Na Out,g Ash
10	Reynolds	760	120	Clay + CaO	2.6	2.6
11	Reynolds	760	120	Clay + H_2O in Combustion Air	2.6	2.7

Grab samples for HCN and HF analysis was withdrawn directly from this manifold. Samples for the continuous monitors for O_2, CO_2, and CO were withdrawn from this manifold and passed through an ice-bath cold trap to cool the sample prior to introduction to the instruments. The continuous monitoring of O_2, CO_2, and CO was performed by standard EPA methods or the equivalent[2]. The monitoring instruments are listed in Table 8. Standard methods were also used to analyze grab samples for HCN[3] and HF[4]. The instruments used to monitor O_2, CO_2, CO, and SO_2 concentrations in the fixed bed combustor effluent were calibrated in accordance with the manufacturers specifications and the recommendations in Volume II of the EPA Quality Assurance Handbook. Multipoint calibrations of each instrument were performed prior to the first test. Zero and span tests were then performed at the start of each days experiment. If these tests deviated from the day to day median by more than 10 percent, the instruments were recalibrated. Calibration and span gases are traceable to the National Bureau of Standards Standard Reference Materials.

All determinations were made such that they provided results that were representative of the matrix in question. The QA objectives of high quality data were met by determining the accuracy and the precision of each determination. The objectives for precision, accuracy and completeness of all the determinations are summarized in Table 9 and Table 10.

The ash from each run was ground and analyzed for CN, F, C and Na_2O. Grab samples of the feed stock for combustion tests were taken from the two head samples furnished by the Aluminum Association. The head samples were prepared by member companies of the association and are considered to be representative of the waste potliner being produced by them. The cyanide in the ash samples was determined using a colorimetric method based on the conversion of CN from an alkaline distillate to cyanogan chloride CNCl, and subsequent reaction with chloramine-T to develop the color. The fluorine in the ash samples was determined by the alizarin sodium monosulfonate thorium nitrate direct titration of the fluoride distilled from the leachate of the fused alkaline sample. Total carbon in the ash samples was determined by the standard Leco combustion apparatus. Sodium in the ash samples was determined by atomic absorption spectrophotometry (AAS). For sodium determination, the ash samples were digested in the appropriate acid and the solution diluted to provide a signal within the calibration range, using the procedure described in the EPA-SW-846 document as the reference method.

TABLE 8. ANALYTICAL INSTRUMENTATION FOR GAS ANALYSIS

Parameter	Instrument
CO	Beckman Model (NDIR)
CO_2	Beckman Model (NDIR)
O_2	Taylor Model (Paramagnetic)

TABLE 9. GAS ANALYSIS QUALITY ASSURANCE OBJECTIVES

Parameter	Completeness (%)	Accuracy	Precision % Rel. Std. Dev.
CO	100	3%	5
CO_2	100	3%	5
O_2	100	3%	5
HCN (Grap Sample)	100	10	5
HF (Grab Sample)	100	10	5

TABLE 10. PRECISION, ACCURACY AND COMPLETENESS OF SOLIDS ANALYSIS

Measurement Parameter (Method)	Reference	Experimental Conditions	Completeness	Accuracy	Precision Std. Dev.
Na - AAS	EPA-SW-846	Acid Digestion	85%	+10%	10%
C - Total	Leco Manual	Leco Analyzer	80%	+15%	10%
CN - Total	EPA-600/4-79-020	Colorimetric	90%	+10%	5%
F - Ionic	Alcoa [914(4-47)]	Titrimetric	90%	+10%	5%

In solids analysis, the response of each analyte using the appropriate analytical technique was compared to the response obtained from calibration standard prepared at concentration within the linear range of the analytical system for that analyte. At least four calibration standards were prepared for each method. The concentration range of these standards was chosen such that it bracketed the anticipated level of the analyte in the sample. If the level of the analyte in some samples fell outside this range, a sample dilution step was implemented and the sample reanalyzed. Calibration curves were constructed by plotting the instrumental response to the analyte vs its known concentration.

The original experimental data for this project are kept in Battelle's Laboratory Record Books Nos. 37519, 38718, and 38975. The records have been reviewed and approved by Battelle's QA Director.

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APPENDIX A

COMMENTS

RELATED TO

BATTELLE'S LABORATORY FEASIBILITY STUDIES FOR THE FLUIDIZED-BED COMBUSTION OF SPENT POTLINING FROM ALUMINUM REDUCTION

September 6, 1983

By

Derek R. Augood

Kaiser Aluminum & Chemical Corp.
Center for Technology
Pleasanton, CA 94566

ADDENDUM TO BATTELLE'S SUMMARY REPORT

ABBREVIATIONS

KACC	Kaiser Aluminum & Chemical Corp.
CFT	Center for Technology
PL	Potlining
CN	(Total) Cyanide
EPA	U. S. Environmental Protection Agency
AA	Aluminum Association

INTRODUCTION

Battelle is conducting laboratory feasibility studies for combusting spent PL. The work is being done for the EPA and the AA.

The work, almost complete in terms of spending, shows, or raises questions concerning:

- Agglomeration
  - Possible low temperature (<700°C) difficulties -- notably with feed.
  - Marked agglomeration and poor reaction in some fixed bed experiments.
- CN Destruction
  - For experiments using relatively dry air, while it initially appeared that little CN was destroyed, re-evaluation of the data shows that CN is ~99% destroyed at ~760°C (while C is simultaneously reduced by ~75%).

When the foregoing initial results were aired on August 25, a request was made for information that the aluminum companies might possess -- information that would supplement the Battelle data.

In separate studies, KACC has developed such information -- some of which has been provided to the Study Steering Committee earlier.

The following Comments, therefore, formalize this KACC information and are provided as an Addendum to Battelle's summary report to the EPA and AA.

SUMMARY AND CONCLUSIONS

1. Comments are provided concerning the feasibility of combusting PL to destroy CN: see INTRODUCTION.

2. While some details are lacking, KACC possesses, or has developed, information relevant to this project.

3. KACC has combusted fine PL on a large scale in a multihearth furnace for several years. As practiced -- at ~675°C for ~2.3 hr -- there is no agglomeration problem.

4. Apart from conceivable low-melting eutectics, agglomeration may be affected by:

   - Incipient NaOH, or water, in the system.

   - Insufficient stirring -- noticeably with $Ca(OH)_2$ or $CaCO_3$ present.

     Gentle agitation, at least, may be required to eliminate agglomeration.

   - Use of $Ca(OH)_2$ as additive -- because this forms a heavy sticky mixture with PL under certain conditions.

5. In general, cyanides are quickly and efficiently destroyed in high temperature ($\geq$800°C) gas systems containing steam.

6. Large scale combustion of PL, along lines indicated in Item 3, shows efficient destruction of Total CN.

7. Supporting results are provided by similar, single stage laboratory combustion experiments.

8. A German disclosure claims destruction of Free- and Total-CN when PL is combusted at 300-500°C.

LITERATURE

A literature search uncovered the following:

Clukey, U.S. Pat. 2,732,283, Feb. 24, 1953 (KACC).

Describes a multiple hearth Herreshoff-type furnace for calcining/combusting PL for ~2.3 hr at ~675°C. The clinker is fed to reduction cells.

The patent mentions:

- Water (steam) added to decompose nitrides and carbides.
- Potential agglomeration problem due to low-melting sodium hydroxide (NaOH). Overcome by operating in a way to first convert NaOH to higher-melting Na_2CO_3.

G. Byer, et al, Environ. Sci. Technol., 1983, 17, 84.

A laboratory study of the kinetics of reaction between HF and CaO for fluoride emission control, when burning PL in a fluidized bed combustor, identifies a relatively low melting eutectic: $NaCa_2FSiO_4$ at 790°C.

German Document of Disclosure 2,628,192, Jan. 20, 1977, (Vereinigte Metallwerke).

Outlines laboratory boat experiments wherein CN, in $\lesssim$1 mm and $\lesssim$3 mm diameter PL, is destroyed in 30-60 min at 300-500°C.

Presumably, ambient (humidity) air is used.

Japanese, Polish and Russian Papers.

Numerous papers discuss combustion of oil (kerosene)-type emulsions with aqueous CN-solutions wherein CN is destroyed. Temperature is generally ~800°C and residence times are short. Specific examples:

Hitachi	1.8 sec; ~810°C; $\leq$7% O_2
Asahi	1.2 sec; 800-1100°C; air ratio 1.25

These processes are gas phase, of course, and steam content is relatively high.

EXPERIMENTAL

Miscellaneous Laboratory Tests (CFT)

Crude "melting point" tests were made some time ago. Small piles of PL, placed on a plate in a laboratory furnace, were observed for onset of melting/slumping.

- The "melting point" of PL appeared to be ~1080°C.
- With ~3% $Ca(OH)_2$ present, the "melting point" was lowered ~100°C.
- With $CaCO_3$ present, major amounts of CaF_2 formed after 4 hr at 800°C.

KACC Reduction Plant

The Clukey process (see above) was operated by KACC for several years beginning ~1952. The process was abandoned when the pots could no longer tolerate impurities in the clinker.

There are no early CN data.

The furnace was operated briefly, twice, in 1982. Data are given in Table 1. Some details are difficult to confirm.

It appears that:

- Apparently, agglomeration was not a problem in operating the furnace.
- Total CN is markedly reduced during PL combustion at ~675°C for 2.3 hr.
- Minor quantities of water (steam) may be required for CN reaction.

Small Herreshoff Tests (CFT)

A one stage Herreshoff furnace has been operated batchwise. The furnace was fired continuously with gas and air. No water was added.

Interim results are given in Table 2. Note that:

- In exposure times up to 7 hr, CN is markedly (~99%) reduced as C is burned away (~51 to 98%).
- Agglomerate, containing black unburned PL, forms in unstirred zones of the furnace when using Ca-additives.
- $Ca(OH)_2$ additive forms a transient, heavy sticky mixture during heat-up. It is speculated that this is due to incipient release of water by the $Ca(OH)_2$. (Could this interact with PL forming NaOH? See above.)

TABLE 1: MULTIPLE HEARTH COMBUSTION OF PL: 1982 DATA

Feed PL ~100% <14 mesh and ~50% <100 mesh

	Total CN wt%	
	Run (1)	Run (2)
PL Feed	0.61 to 1.31	0.5 to 1.9
Clinker	0.002	0.0006 to 0.006

During these runs, C decreased from ~27 to ~3.5 wt %.

Auxiliary gas was used as fuel during startup and was apparently turned down later.

It is unclear whether or not water was fed to the system.

A couple of off gas analyses during Run (2) indicated 2.0 to 4.2% H_2O.

TABLE 2: SINGLE STAGE HERRESHOFF FURNACE

Batch Tests: Interim Results

Feed			Furnace		Product		~% Reduction (h)	
PL Source(a)	PL Size	Additive	°C(c)	hr(d)	Color	Agglomeration	C	CN
I	<14 mesh	None	~835	3.2	Grey	None	79.7	99.5
I	1.65 to 3.2 mm			6.0	"	"	50.8	99.2
III	4.7 to 9.4 mm			5.5	White & brown	Considerable attrition(e)	--	(i)
II	0.30 to 1.65 mm		~945	5.8	"Salt & pepper"	None	92.9	99.2
II	——"——	$Ca(OH)_2$ (b)		5.7	Yellow	Yes(f) Sticky at ~570°C(g)	98.5	99.0
II	——"——	$CaCO_3$		7.4	Brown	Yes(f)	97.9	98.8

(a) I, II and III different lots.
I = Retain sample from Run (2) at reduction plant. See Table 1.
II = Fraction from sample of ~3 ton lot.
III = Fraction from ~40 kg sample of >5 cm dia. lumps.

(b) Size <10 mesh; 91.9% $Ca(OH)_2$ and 8.2% $CaCO_3$ based on Ca and CO_3 analyses. Also analyzed 0.2% Si.

(c) Bed temperature, measured by thermocouple at bottom of bed, read low (~155°C?) because of relatively stagnant layer of solids covering thermocouple. Quoted temperature is estimate based on measured temperature, temperature of gas and some pyrometer readings. Gas temperature often ~1000°C.

(d) Time from start at room temperature. System took 1.5 to 2.0 hr to reach target temperature -- except $CaCO_3$ case which took 3.7 hr.

(e) Possibly due to crushing between rabble arm and wall or charge.

(f) Hard agglomerate in dead, unstirred zones -- particularly in grooved/worn areas on sides of furnace. Material contained unburned PL.

(g) Charge became sticky and heavy around 570°C (estimate) during heat up. Material "dried out" after ~2.3 hr and remained granular. A few ~10 mm dia. pieces in product, but hard agglomerate in dead zones -- see (f) above.

(h) Percentage ratio given by [Δ Wt % (PL - Clinker)]/[Wt % PL].

(i) Analysis of clinker shows CN at low level comparable with other clinkers.

ADDENDUM TO BATTELLE'S SUMMARY REPORT

ON

LABORATORY FEASIBILITY STUDIES FOR THE FLUIDIZED-BED COMBUSTION OF SPENT POTLINING FROM ALUMINUM REDUCTION

SEPTEMBER 13, 1983

BY

RON N. DRAKE

GA TECHNOLOGIES INC.
SAN DIEGO, CALIFORNIA 92138

Small scale laboratory experiments were performed at GA Technologies Inc. to obtain preliminary indication of the physical behavior of spent cathode waste and ash during combustion. Experiments were carried out in a one inch diameter tube furnace and a small laboratory muffle oven, using samples of potlining supplied by Martin Marietta and Alcoa.

The tests are summarized in the attached table. Various combinations of first and second cut potliner, potliner ash and additives were observed during heating to determine at which temperatures the materials would cake, agglomerate and fuse. Additives used were reagent grade $CaCO_3$, limestone and calcined limestone.

Conclusions and observations are as follows:

- Based on observed agglomeration behavior, FBC processing of both MML and Alcoa supplied potlining should be possible if bed temperature is maintained below 900°C.
- Additives effectively increase the ash fusion point but promote low temperature (790-825°C) caking of test materials.
- Low temperature caking of the material seems transitory and is alleviated by gentle agitation.
- Pre-calcined limestone is significantly better than reagent grade $CaCO_3$ or limestone additives from the standpoint of low temperature caking behavior.

Although tube furnace and muffle oven combustion tests do not model fast fluid bed behavior, such tests do provide a worst case environment for producing caking, agglomeration, and fusion. Evaluation of similar tests by experienced personnel at GA has provided the basis for planning and execution of several successful pilot plant demonstration runs with other materials exhibiting a high potential for agglomeration and melting.

SUMMARY OF LAB SCALE POTLINING COMBUSTION EXPERIMENTS AT GA

Test	Source	Cut Mix, % (1) 1st	2nd	Pretreatment	Charge Mix	Combustion	Temp., °C	Wt. Loss, % (2)	Comments
1	MML	100	-	Ground -200 mesh	70% PL 30% $CaCO_3$	Tube Furnace (3)	850-960	41	Soft cake @ 870°C, (Uniform soft friable) Aagglomeration @ 880°C Unsuitable for FBC @ 960°C
2	MML	100	-	Ground -200 mesh	50% PL 50% Limestone	Tube furnace	820-880	25.3	Very slight caking @ 830 Material well behaved on stirring @ 880°C
3	MML	50	50	Ground	100% PL	Muffle Oven	1100	31.3	Forms brittle vitreous melt
4	MML	50	50	Ground -200 mesh	65% PL 35% Calcined	Muffle Oven	1000	19.6	Forms candy like mass which hardens on cooling
5	MML	50	50	Ground -200 mesh	50% PL 50% Calcined Limestone	Tube Furnace	840-950	33.3	Well behaved easily stirred ash suitable for FBC
6	MML	50	50	Ground -200 mesh	35% PL 65% Limestone	Muffle Oven	1100°C	23.6	Easily stirred at temp but forms agglomerates on cooling
7	MML	50	50	Ground -200 mesh	100% PL	Tube Furnace	700-840	29.3	Form light grey, loose well behaved ash suitable for FBC
8	MML	50	50	Ground -200 mesh	80% PL 20% Limestone	Tube Furnace	830-960		No noticeable agglomeration @ 840°C. Some fusion at boat wall but bulk material loose, well behaved and suitable for FBC @ 960°C

SUMMARY OF LAB SCALE POTLINING COMBUSTION EXPERIMENTS AT GA
Page 2

Test	Source	Cut Mix, % (1) 1st	2nd	Pretreatment	Charge Mix	Combustion	Temp., °C	Wt. Loss, % (2)	Comments
9	MML	50	50	As supplied minus 1/4 in.	100% PL	Muffle Oven	700-850	29.3	Loose well behaved ash suitable for FBC
10	ALCOA	As supplied		As supplied minus 1/4 in.	100% PL	Tube Furnace	729-950	37	@ 750°C well behaved @ 850°C well behaved @ 860°C some sticking at boat wall, bulk material suitable for FBC @ 880°C No Change @ 900°C Sticking at wall, some caking, bulk material still suitable for FBC @ 950°C Melt at boat wall, bulk material fused and agglomerated, not suitable for FBC
11	ALCOA	As supplied		As supplied minus 1/4 in.	100% PL	Muffle Oven	300-800	38	Slight caking from 770 to 800°C, but bulk material suitable for FBC
12	ALCOA	As supplied		Ash from Test 11	80% Ash	Muffle Oven	820-950	-	@ 825 Some sticking at crucible wall, bulk material loose and well behaved. suitable for FBC @ 850 No change @ 875 No change @ 900 No change @ 950 No change

SUMMARY OF LAB SCALE POTLINING COMBUSTION EXPERIMENTS AT GA
Page 3

Test	Source	Cut Mix, % (1) 1st	2nd	Pretreatment	Charge Mix	Combustion	Temp., °C	Wt. Loss, % (2)	Comments
13	ALCOA	As supplied		Ash from Test 11	65% Ash 35% Calcined Limestone	Muffle Oven	875-1100	-	@ 890 Some attack on crucible wall, slight caking but easily stirred, suitable for FBC @ 1100°C, Some melting at crucible wall, moderate caking, borderline for FBC

(1) 1st Cut primarily cathode block, 2nd Cut primarily insulating materials
(2) Wt. loss on potlining basis
(3) Plant air used as sweep gas, ambient humidity